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Original research paper

Study on experiment conditions of marine shale gas seepage law☆

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Abstract

In order to discover the conditions suitable for testing shale gas seepage law, marine shale gas cores were taken from southern China. Samples were tested by using the differential pressure-flow rate method with actual gas under two modes (e.g. constant confining pressure and constant net confining pressure). Moreover, influences of the different confining pressure modes on the experimental results were analyzed. The results show that under constant confining pressure or constant net confining pressure mode, the gas seepage law curve has two sections. One is the curve section and the other is the pseudo linear section. Features of non-linear seepage were observed with the inflection points of 1 MPa and 1.3 MPa, as well as the average permeability damage rate of 52.41% and 40.56% respectively. The slip effect generated different influences. In the constant confining pressure mode, the change of injection pressure may cause stress sensitivity, which is not consistent with the actual situation in the reservoir development. The influence of the slip effect on seepage law was more substantial than stress sensitivity under the condition of low effective stress. In the constant net confining pressure mode a complete seepage law curve was obtained to simulate the seepage of the actual reservoir in a certain extent. The confining pressure effect had an insignificant influence on gas seepage. Comprehensive analysis shows that net confining pressure mode is the best way to test the seepage law of marine shale gas core in southern China.

Keywords: Seepage law; Constant confining pressure; Constant net confining pressure; Slip effect; Effective stress; Confining pressure effect

1. Introduction

Shale gas is a type of non-conventional gas, which is of extreme importance. Its development has been paid ever more attention and the exploration in China is also in its formative years [1–13]. The experimental method is different from the conventional gas because of its special structural features of bedding, joints, microfractures [4,5,8,9]. Due to better lithology and larger permeability of core in conventional gas, the constant confining pressure mode is generally adopted to test the gas seepage law [14], without considering the stress sensitivity caused by the effective stress changes [15–21]. It is necessary to consider the influence on shale reservoirs with special physical properties, because the change of effective stress may lead to the destruction of the bedding and cracks in shale; which could influence test results of the shale seepage law. Thus, the test of shale gas seepage law has become an urgent problem needing to be solved. In this paper, the suitable test method to hold the experimental pressure was adopted to select the best condition for testing the gas seepage law of the marine shale gas cores [22–25]. Eventual results opt to provide a useful reference for China's shale gas research and development.

2. The characteristics of shale gas reservoir

The shale gas reservoir in Longmaxi Formation, southern China, is buried deep and types in black laminar clay (shale),

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lamellar silty clay (shale) and lamellar carbonate substance (gray or dolomitic) clay (shale), developed graptolite. Analysis of the lithological properties of shale gas reservoir shows that the sizes of main (major) nanopores were in the range of 5—200 NM, the porosity was in the range of 0.58%—4.27%, both were averaged to1.26%.The permeability was in the range of 0.00001—0.93 $\times$ 10^{-3} μm^2, averaged value was 0.0067 $\times$ 10^{-3} μm^2. The content of the organic matters was commonly lower and was averaged to 2.46%.

The X-ray diffraction analysis showed that the total amount of the clay minerals were relatively high with an average of 53.39%. Followed by quartz laminar or dispersed, with an average content of 29.15%, particle diameter was in the range of 0.03—0.05 mm, and with the high content of quartz lead to the result of high brittle minerals, which is good for making cracks and exploiting gas. Moreover, calcite and feldspar in clay mineral have an average content of 5.46% and 4.93%, respectively. Others, such as dolomite, gypsum, pyrite, and other minerals all together had only an average content of less than 5%.

The physical property of the reservoir was poor. The core analyzed the interformational pores, intragranular pores, and microfractures were developed and followed by the construction fractures as well as the intercrystalline micropores. The interformational pores included illite, chlorite flakes pores, pore mica flakes, raspberries pyrite intergranular pores, and more. The intragranular pores included quartz and intergranular solution hole that was caused by the dissolved feldspar. The pore diameter was in the range of 100 nm—50 μm, and was frequently had micro-cracks (width of 2—5 μm). The microfractures were mostly the fractures in Erie stone layer or the microfractures between brittle clay minerals and quartz; having a width of 5—20 μm. The maximum throat radius was 0.033 μm in average. The mean value of the throat radius was 0.010 μm. Mainstream pore throat radius was 0.0038 μm, in which microporous and the micro-throats belong.

3. Experiment

3.1. Apparatus and equipment

The seepage experimental device selected was a US core company's Autoflood (AFS300TM) flooding evaluation system. The high-pressure nitrogen tank provided the injection gas, and it controls the pressure by adjusting the regulator. The data acquisition system collected various data through the pressure system acquisition automatically. In order to simulate the characteristics of formation stress, the triaxial core holder was used in the laboratory, it measured the gas via a soap bubble flow meter. High-precision multi-stage piston displacement pump (Teledyne Isco 100 DX) controlled the confining pressure system. Pressure was measured by a high-precision digital sensor (DXD), whilst using a high linearity differential pressure sensor (validyne models) to accurately acquire the differential pressure at both ends of the core. The entire device was placed in a thermostat, and the temperature was set to 0—180 °C (Fig. 1).

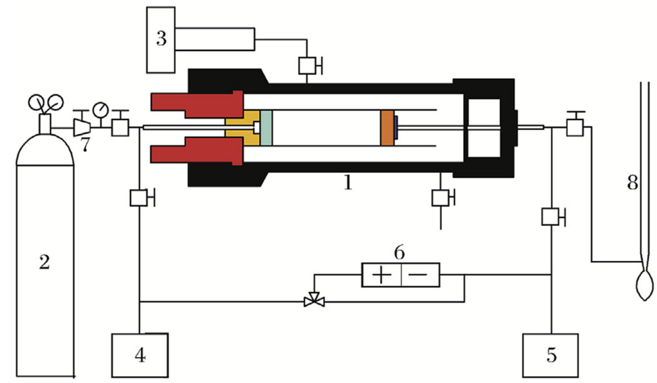

Fig. 1. Schematic diagram of the experimental system. 1- Triaxial core holder, 2- High pressure gas cylinder, 3- Multi-steps pump drive, 4, 5- Up-downstream pressure sensor, 6- High linearity differential pressure sensor, 7-Pressure regulating valve, 8-Soap bubble flow meter.

3.2. Project and steps

The experiments used an intact core from the Well LongShan-1, which served as the representative of the Qiongzhusi reservoir. The length of the core was 5 cm and the diameter was 2.5 cm. It used the on-site gas as the injection fluid. The experiment used the "differential pressure-flow rate method" with constant temperature and atmospheric pressure. According to different confining pressure control methods, the experiment was intended for constant confining pressure mode and constant net confining pressure mode. In comparison, the pressure for the constant confining pressure mode was set to 4 MPa, and the pressure in the constant net confining pressure mode was set to 3 MPa. This ensured that the core's effective pressure gave only a slight difference between the two modes while the confining pressure or the injection pressure changes.

3.2.1. Constant confining pressure mode

The constant confining pressure was 4 MPa, and the pressure points used in the injection pressure experiment were 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.5, and 2.0 MPa. This helped determine the stable flow under different injection pressures.

The experiment steps are as follows: (1) The core was dried for 48 h, then it's length, diameter, and porosity was measured through the Klinkenberg permeability via regression method. (2) The core was placed in the core holder, this helped connect the process, after which the initial value of the instrument was set to zero. The confining pressure was slowly set to four MPa, but there was no evident change. (3) In accordance to the predetermined injection pressure on-site gas was injected into the core for 30 min; then the soap bubble flow-meter was used to collect the time points thrice at the same volume continuously. The size of the three time points are as follows: "big-small-big" or "small-big-small"; and the difference between the two adjacent points was less than 0.03 s, then the stable flow was considered. It was necessary to extend the flowing time and to re-determine everything until the flow was stable; then record the pressure and flow. (4) Lastly, the next pressure points were replaced and the third step was repeated to determine all the pre-determined pressure points which ultimately finished the experiment.

3.2.2. Constant net confining pressure mode

During the experiment, the confining pressure was always set to 3 MPa, which is higher than the injection pressure Therefore, once the inlet pressure was adjusted, the confining pressure was adjusted as well. According to the pre-determined injection pressure, the injection pressure was set to 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.5, and 2.0 MPa. This was done to determine the stable flow under different injection pressure.

The experiment steps are as follows: (1) The core was dried for 48 h, then its length, diameter, and porosity was measured through the Klinkenberg permeability. (2) The core was placed in the core holder, this helped connect the process, after which the initial value of the instrument was set to zero. The confining pressure was slowly set to three MPa. (3) Afterwards, set the holder's inlet pressure as the injection pressure and confining pressure were adjusted; the confining pressure should always be 3 MPa higher than the injection pressure. (4) According to method aforementioned use the soap bubble flow-meter to measure the gas flow until the flow was stable, then record the pressure and flow. (5) Once the injecting pressure and the confining pressure were adjusted per point, determine all the pre-determined pressure points which finished the experiment.

4. Result analysis

The experiments were conducted based on the programs mentioned above. The experimental determination of each point was able to pinpoint at what pressure stable gas flow. As a result the adsorption—desorption equilibrium was established. This equilibrium does not affect the flow of a stable supply source. During data processing and analysis, the microscale effect was ignored.

4.1. Constant confining pressure

The constant confining pressure was Four MPa, and the injection pressure was increased during the experiment. Results are shown in Fig. 2. The curves showed that the gas flow rate increased significantly with the increase of injection pressure. The curve shapes of pressure squared difference and the flow were convex whose tangent slopes were decreased in the case of low driving pressure. Whenever the drive pressure was higher than the gas flow rate, the curve shapes of the relationship between the pressure squared the difference and flow were quasilinear. Thus, the flowing law curve has the bending part and the quasilinear part. For example, the curve whose permeability was 0.2968×10^{-3} $\mu m^{2,}$ and the injection pressure was less than one MPa was bent and when the injection pressure was more than one MPa it was quasilinear. In Fig. 2, the bending parts of the curves were not clear with low

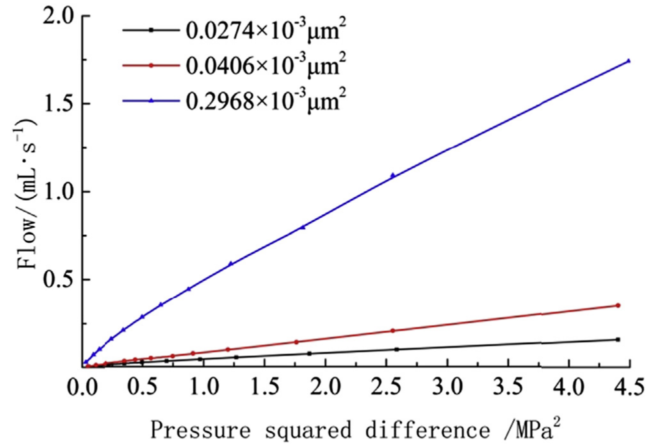

Fig. 2. Gas seepage law under the condition of constant confining pressure.

permeability. The curve shapes of the relationship between the pressure squared difference and the flow were quasilinear.

The lower permeability core was denser and was harder to be compressed or to deform in low effective pressure. When the confining pressure was constant and the injection pressure was low, the effective pressure of the core is high. Thus, deformation of the core's skeleton was high. The degree of micro-fractures closure and pore compression was large and the percolation effect was poor. As the injection pressure increased, the effective pressure of the core decreases, and the stress deformation of the core matrix decreases as well. However, the deformed portion of the skeleton recovers and the percolation effect becomes better. If the impact of the changes in effective stress was considered, the percolation effect will get better with the increased injection pressure. However, the permeability was decreased in the experiments. Therefore, the influence of the slippage effect cannot be ignored. In Fig. 3, the injection pressure of one MPa is an inflection point. In the interval., the pressure was lower than one MPa causing the permeability to decline faster because the amplitude of cracks were closing and the degree of the pore compression was greater. In the interval that the pressure was

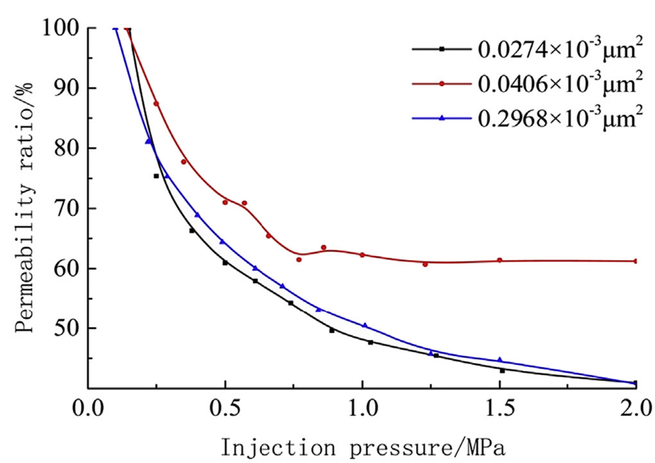

Fig. 3. Permeability change law under the condition of constant confining pressure.

higher than one MPa, the cracks and pores were harder to be compressed, and the influence of the slippage effect was weaker and the permeability hardly changed. Whenever the gas flow was in a low pressure, the effects of slippage were evident [14]. When the injection pressure increased, the slippage effect was decreased. The seepage effect will be slightly worse and the permeability will gradually reduce. At the same time, the rock matrix effective stress decreased; this strengthens the flow. The effects of the two counteract mentioned and the last result was that the percolation effect gotten worse. Therefore, the slippage effect was more important than the effective pressure changing in the case of the low effective pressure. The seepage Law was mainly influenced by the slippage effect. The corresponding experiment showed that the seepage flow deteriorates with the increased injection pressure, and the macro performance reduced permeability.

This experiment was conducted on the condition in which the injection pressure is high. If the injection pressure was decreased, the consequence of the slippage effect will increase the effective pressure. 'The results of both offset will increase the permeability; the result of the slippage effect will be much higher than the impact of changes in the effective stress. Therefore, if the unrecoverable portion of the compressed rock was ignored, the gas flow curve will not be affected by the changing process of the injection pressure.

4.2. Constant net confining pressure

According to the net confining pressure of three MPa, once the inlet pressure was adjusted, the confining pressure was adjusted as well. The results as shown in Fig. 4. The nonlinear character of the flow curve was not very obvious, it was divided into two stages when the injection pressure got to 1.3 MPa (the pressure difference is 1.95 MPa^2). The feature was not obvious below the pressure, but when the pressure difference was less than 0.5 MPa^2, the feature became relatively obvious. Whenever, the injection pressure was more than 1.3 MPa, the flow curve has the similar characteristics of linearity. Characteristic of nonlinear low permeability core was not very clear.

According to the effective stress theory, when the effective stress was constant, the deformation degree of the rock body remains the same. Thus, the seepage flow will not change. The seepage flow of shale gas was influenced by the slippage effect. As Fig. 5 shows, the range of permeability variation was more than 30%, especially the cores whose permeability was 0.1030×10^{-3} μm^3. When the injection pressure was close to two MPa, the penetration rate was more than 50%; which shows the effect of the change of the confining pressure. Still referring to Fig. 5, the change of the permeability curve was divided into two stages with the pressure of one MPa. When there was an increase in injection pressure, the change of the permeability gradually slowed down; this also proved the conclusion wherein the influence of the slippage effect with a low pressure was greater than in a high pressure. When the injection pressure was 0.2 MPa, the cores with the permeability value of 0.0113×10^{-3} μm^2 and 0.0520×10^{-3} μm^2 increases; which is related to the rock heterogeneity. The higher the permeability is, the greater the change it undergoes. When the core's permeability was 0.1030×10^{-3} μm^2, the range of the permeability variation was wider than the other two cores' slightly small permeability because of the shale particularity, bedding, and joints that led to a mass of micro cracks in the rock; this proved that micro cracks influenced the permeability. The micro cracks became easy to be enclosed within a low effective stress.

Having the condition of the constant net confining pressure, the confining pressure changed with the injection pressure. The core within the holder was different from that in the reservoir. Overburden pressure was vertically acting on the reservoir, and the rock in the vertical direction was affected by the pressure of the upper layer and the pore pressure. The same pressure, but of horizontal direction, was equal in the original reservoir. However, the force was offset. When the core was fixed in the core holder, which simulated the overlying pressure of the center axis of the core, the whole core had an equal pressure body. The surrounding pressure was equal in both of

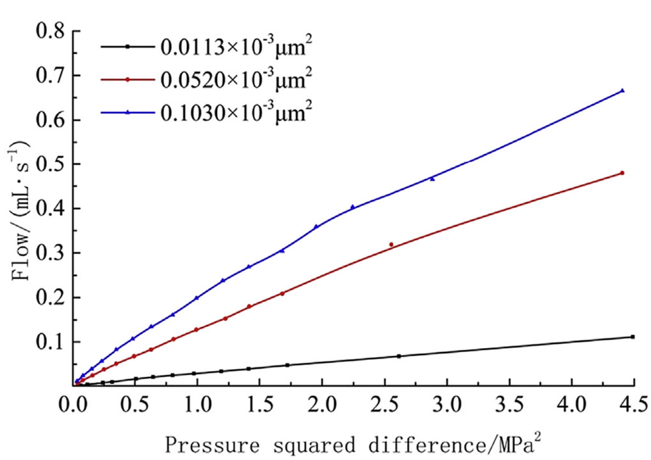

Fig. 4. Gas seepage law under the condition of constant net confining pressure.

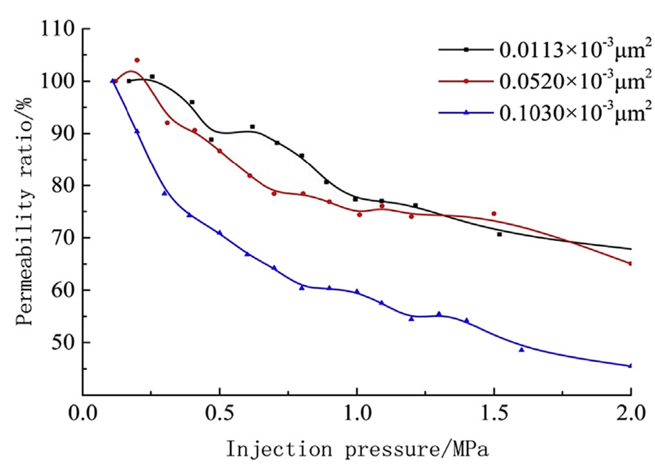

Fig. 5. Permeability change under the condition of constant net confining pressure.

the core's internal and external; not to mention, the value was the same as the confining pressure.

The difference between the confining pressure and the injection pressure was used to serve as the net stress, and the pressure gradient between the inlet and the outlet was neglected. In fact, the net stress value was changed in the direction of the length. The difference between the confining pressure and the inlet pressure was insignificant. On the condition of the constant net confining pressure, the confining pressure increased with the increase of the injection pressure, and the increase of the inlet pressure made the influence of the slippage effect insignificant as well. On the other hand, considering the increase of the confining pressure that resulted in the re-distribution of the confining pressure, whenever the injection pressure increased, the pressure gradient and the outlet net stress were increased. The deformation degree was increased from the inlet to the outlet. Therefore, even if the net stress was constant in the conventional sense, it couldn't assure us that the seepage flow remains constant. As the injection pressure increase, the permeability value will be lower and lower.

On the condition of the constant net confining pressure, factors that influenced the gas flow were slippage and confining pressure effects. When the injection pressure increased, the influence of slippage effect weakened, and the increase of the confining pressure caused subsequent pressure effects; results showed that the seepage flow was worse. Two kinds of effects were superimposed on each other. This exacerbated the gas seepage flow.

5. Problem and reflection

The two kinds of experiment method, namely the constant confining pressure and the constant net confining pressure, cannot eliminate the effects of the confining pressure, but they do have different influence ranges. Fig. 6 shows the permeability damage rate comparison chart in relations with the two modes. It can be seen that the permeability damage rates were different at the similar permeability core on different confining pressure conditions. The permeability damage rate of the constant net confining pressure should have been inferior to the constant confining pressure. The foregoing analysis showed the two kinds of confining pressure. Gas seepage flow was influenced by the slippage and the confining pressure effect, the two effects weakened each other when the confining pressure was constant; superposition of the two kinds of interactions at the condition of the constant net confining pressure. The confining pressure effect on gas seepage of constant confining pressure was greater than the constant net confining pressure with the same slippage effect. It can be concluded that, the constant net confining pressure should have had a large permeability damage In Fig. 6, we can observe that the slippage effect was different in the two modes, the influence of the slippage effect of the constant net confining pressure should have been inferior to the effect of constant confining pressure condition, especially when the injection pressure increases the former influences on the rates were much less than the latter. Thus, we can infer that the changes of the confining pressure were influential to the slippage factor.

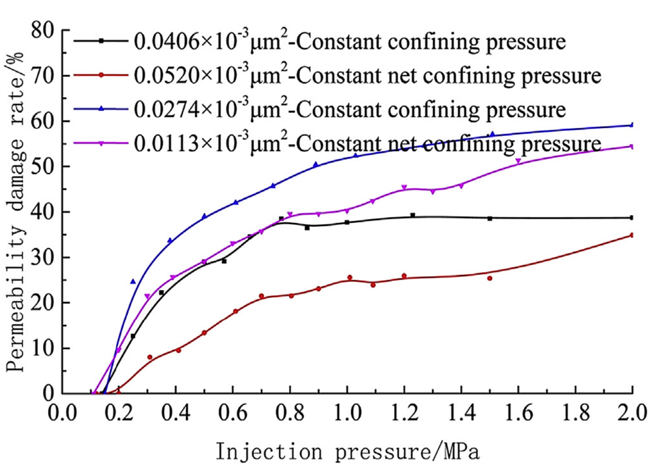

Fig. 6. The permeability damage rate with different confining pressure mode.

5.1. Seepage characteristics test

There was a great depth of the actual shale reservoir, the overburden and formation pressures were high. However, it was not necessary to consider the influences of the slippage effect in the original net stress case. The pressure gradient was low at the horizontal direction in the initial exploitation; the decrease of the permeability was not obvious. As the exploitation goes on, especially in the middle and later periods, the scope of the gas expands and the range of the current net stress increased largely relative to the original net stress near the production well. The permeability was greatly reduced, and the damage was great as well. Nevertheless, for a slight seepage section in the reservoir, the pressure gradient with tiny variations which can be ignored in the different stages of the exploitation and the effective stress can be thought as a constant during the period.

When measuring the gas seepage flow rule in the laboratory at a relatively small injection pressure, comparisons to the constant confining pressure had less slippage effect and permeability damage. The constant net confining pressure mode was relatively insignificant. Laboratory experiments usually select cores with a length of 5–8 cm. Its net stress change can be ignored because of the small size compared to the entire formation. The points in the seepage rule curve, which measured in a constant confining pressure has different net stress, especially when the pore pressure drops greatly. The impact of the stress on the net flow was more significant that it even leads to serious stress sensitivity; due to the tiny size of it in the experiment, it also exaggerated the impact of the effective stress. The experiment of the constant net confining pressure relatively weakened the impact of the effective stress. Hence, the resulting curve was mainly affected by the slippage effect, and the same flow curve has a similar slippage effect and confining pressure. It was consistent with the real reservoir development.

Based on the analysis above, it was better to measure the gas flow rule when the net confining pressure was constant. A simulation of the gas flow in the real reservoir situation in a period would be a meaningful reference for the actual development.

5.2. Confining pressure stress sensitivity

The conventional confining pressure stress sensitivity experiment kept the inlet pressure at a constant value together with the gradual increase of the confining pressure. The shale stress sensitivity experimental considered the actual high pressure formation. The experiment method eliminated the influence of the slippage effect to ensure that the only influencing factor to the permeability was the confining pressure. Therefore, it was suggested to make a high injection pressure. However, to accurately simulate the actual formation of stress conditions, the best way was to set the confining pressure as a constant value. The confining pressure was set as the actual formation overlying the pressure constantly. The injection pressure from the actual formation pressure was set to a lower quantity; certain pressure points were abandoned, but the net stress laboratory point was ensured to be enough.

6. Conclusions

In this paper, the experimental study, according to the plan was carried out by using a real core from the reservoir, and an optimum condition was chosen to determine the shale gas seepage law. The conclusions are as follows:

(1) Shale gas seepage flow increased with the increase of the injection pressure. The flow curve was divided into curve segments and quasilinear section; with a characteristic of non-linear. In both confining pressure mode, the injection pressure increased. The magnitude of the permeability reduced slowly, but the rate of the permeability damage was 30%, in different modes of the confining pressure the slippage effect varies widely; the slippage effect has greater influence on the condition of constant confining pressure.
(2) In the condition of constant confining pressure, the shape of the flow curve was changed from a curve section of convex direction flow of a curve of the quasilinear feature with increasing pressure. Under the condition of a low effective stress, the flow rule was mainly influenced by the slippage effect, and the change of the injection pressure does not affect the flow curve; the flow curve was mostly influenced by the confining pressure, and the appropriate experimental stress sensitivity.
(3) In the condition of constant net confining pressure, the seepage curve was divided into two segments at the injection pressure of 1.3 MPa, and the character of the nonlinearity was not obvious. The higher the permeability of the core, the greater the microcrack contribution. The influence of the confining pressure could not be eliminated.
(4) It was better to use the mode of constant net confining pressure in measuring the flow rule of gas. This mode simulated the actual reservoir flow conditions in a period of time and provided a meaningful reference to the reservoir.

Foundation item

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Conflict of interest

The authors declare no conflicts of interest.

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